

Synthesis of Tantalum- and Niobium Iodides by
Interaction of $TaCl_5$ and $NbCl_5$ With Al_2J_6 and SiJ_4

M. I. Kalinin

SUBMITTED: September 17, 1958

68217

S/078/60/005/02/003/045
B004/B016



Card 3/3

NISHEL'SON, L.A.; SHRYAKOV, G.V.

Systems: $\text{SiCl}_4 - \text{PCl}_3$, $\text{SiCl}_4 - \text{POCl}_3$, and $\text{SiCl}_4 - \text{PCl}_5$.
Zhur. neorg. khim. 5 no.5:1139-1145 May '60. (MIRA 13:7)

1. Moskovskiy institut teoreticheskikh metallov im. M.I. Kalinina.
(Silicon chloride) (Phosphorus chloride)
(Phosphoryl chloride)

S/076/EO/C05/C07/035/043/XX
3004/3060

AUTHORS: Nisel'son, L. A., Chernyayev, V. K.

TITLE: $SiI_4 - BI_3$ and $SiI_4 - Al_2I_6$ Systems

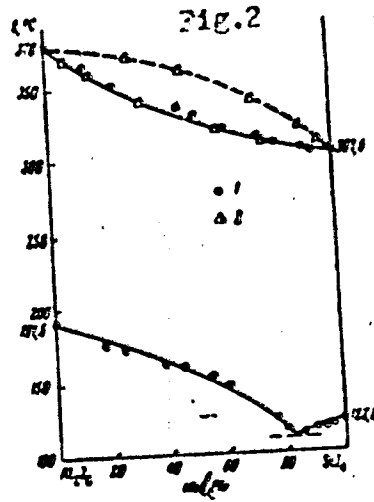
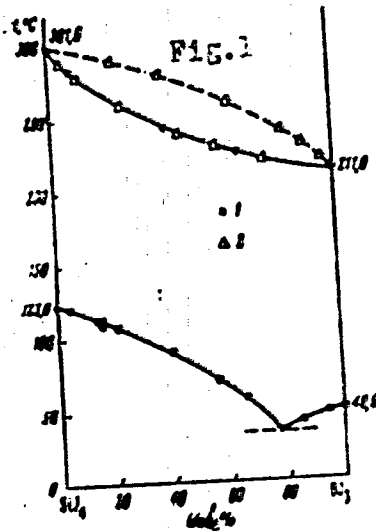
PERIODICAL: Zhurnal neorganicheskoy khimii, 1960, Vol. 5, No. 7,
pp. 1564-1566

TEXT: In view of the fact that volatile iodides are used to produce high-purity elements, the authors examined the phase equilibria in the $SiI_4 - BI_3$ and $SiI_4 - Al_2I_6$ systems. The phase equilibrium between crystals and liquid was determined by a method described in Ref. 1, and the boiling point according to Ref. 2 at 760 torr. Results are given in Tables 1,2 and in Figs. 1,2.

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$\text{SiI}_4 - \text{BI}_3$ and $\text{SiI}_4 - \text{Al}_2\text{I}_6$ Systems

S/078/60/005/007/035/043/XX
B004/B060



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$\text{SiI}_4 - \text{BI}_3$ and $\text{SiI}_4 - \text{Al}_2\text{I}_6$ Systems

S/071/60/005/007/035/043/XX
B004/B060

Eutectics appear in both systems. A linear dependence was found between the logarithm of the molar SiI_4 fraction and the reciprocal value of absolute temperature. The thermal effects calculated from the tangent of this straight line lie near the melting heats of SiI_4 and Al_2I_6 . The systems, therefore, follow the Schröder equation. The Raoult law holds for both systems. This was confirmed by measuring the pressure of saturated vapor of pure SiI_4 . Table 3 gives the boiling points of Al_2I_6 between 148 and 854.5 torr, and of SiI_4 between 105.5 and 880.0 torr, determined by means of a Sventoslavskiy ebullimeter. The authors point to the possibility of calculating the phase equilibrium between liquid and vapor from the data relative to the phase equilibrium crystal - liquid, and vice versa, on the basis of the activity coefficients, provided the system does not deviate too much from an ideal one. There are 2 figures, 3 tables, and 2 references: 1 Soviet, 1 US, and 1 German. ✓

SUBMITTED: March 12, 1959

Card 3/4

$\text{SiI}_4 - \text{BiI}_3$ and $\text{SiI}_4 - \text{Al}_2\text{I}_6$ Systems

S/078/60/005/007/035/043/XX
B004/E060

Legend to Fig. 1: Phase equilibria in the $\text{SiI}_4 - \text{BiI}_3$ system

Legend to Fig. 2: Phase equilibria in the $\text{SiI}_4 - \text{Al}_2\text{I}_6$ system

To both Figs.: 1) points determined experimentally
2) calculated by Raoult's equation

✓

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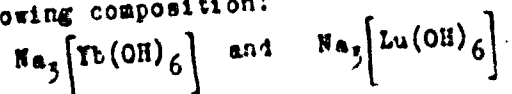
NISL'SON, L.A.

Interaction between NbCl_5 and PCl_3 , and between
 SbCl_5 and PCl_3 . *Zhur. Neorg. Khim.* 5 no.7:1634
J1 '60. (MIRA 13:7)
(Niobium chloride) (Tantalum chloride)
(Thiophosphoryl chloride)

S/078/60/CO5/009/0.8/040/XX
B017/B058

AUTHORS: Ivanov - Emin, B. N. and Nisel'son, L. A. ✓
TITLE: Amphoteric Properties of Ytterbium^{III} and Lutecium
Hydroxides
PERIODICAL: Zhurnal neorganicheskoy khimii, 1960, Vol. 5, No. 9,
pp. 1921 - 1923

TEXT: The alkali salts of the hexahydroxo compounds of ytterbium and lutecium were synthesized. Ytterbium and lutecium oxides with a purity of 99.95% were used as starting materials. The amphoteric hydroxides were treated with a concentrated sodium hydroxide solution for 48 hours in an autoclave at 180 - 200°C. The isolated compounds have the following composition: ✓



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Amphoteric Properties of
Ytterbium and Lutecium Hydroxides

S/078/60/005/009/018/040/XX
B017/B058

The hydroxo compounds of ytterbium and lutecium decompose in water to form sodium hydroxide and the corresponding hydroxides. Through heat treatment of the hydroxo compounds of ytterbium and lutecium at

320 - 350°C, these compounds lose two water molecules. Sodium meta-ytterbiate NaYbO_2 and sodium meta-luteciate NaLuO_2 are produced by heating equivalent mixtures of the corresponding lanthanide oxides with Na_2CO_3 at 900°C. There are 2 figures, 1 table, and 6 references: 2 Soviet, 3 German, and 1 Czechoslovakian.

SUBMITTED: June 19 1959

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03777

S/078/60/005/009/028/0A0/XX
B017/B058

11.4100

AUTHORS:

Ivanov-Emin, B. N., Nisel'son, L. A., Larionova, L. Ye.

TITLE:

Study of the Behavior of Some Fluoro Gallates of Alkali
Metals in Aqueous Solutions

PERIODICAL:

Zhurnal neorganicheskoy khimii, 1960, Vol. 5, No. 9.
pp. 1993-1995

TEXT: The fluoro gallates of potassium, rubidium and cesium were studied in aqueous phase by means of electrical conductivity measurements. The molecular electrical conductivities found for the compounds $[\text{GaF}_5 \cdot 3\text{H}_2\text{O}]$, $\text{K}_2[\text{GaF}_5 \cdot \text{H}_2\text{O}]$, $\text{Rb}[\text{GaF}_4 \cdot 2\text{H}_2\text{O}]$ and $\text{Cs}[\text{GaF}_4 \cdot 2\text{H}_2\text{O}]$ are given in Fig. 1 and Table 1. The determination was made at 20°C . It follows from the studies of the conductivity of fluoro gallates of potassium rubidium and cesium that the complex anion decomposes in aqueous solution according to the reaction schemes: $\text{K}_2[\text{GaF}_5 \cdot \text{H}_2\text{O}] \rightarrow 2 \text{KF} + \text{GaF}_3 + \text{H}_2\text{O}$ and $\text{Cs}[\text{GaF}_4 \cdot 2\text{H}_2\text{O}] \rightarrow \text{CsF} + \text{GaF}_3 + \text{H}_2\text{O}$.

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85599

Study of the Behaviour of Some Fluoro Gallates of Alkali Metals in Aqueous Solutions S/078/60/005/009/028/040/XX B017/B058

respectively. When diluting the gallium fluoride solutions, hydration and dissociation of the hydrated gallium fluoride molecule sets in according to the reaction scheme: $[GaF_3 \cdot 3H_2O] + H_2O \rightarrow [GaF_2 \cdot 4H_2O]^+ + F^-$. The dissociation rises slowly with increasing dilution and the molecular conductivity attains the valence of a two-ion electrolyte only at a dilution of $V \approx 700 - 1000$ /mole. The authors mention I. V. Tananayev and N. V. Bausova. There are 1 figure, 2 tables, and 5 references: 4 Soviet and 1 British.

SUBMITTED: June 6, 1959

Card 2/2

S/078/60/005/000/022/080/XX
8017/8058

AUTHORS: Ivanov-Emin, B. N., Nisel'son, L. A., Greksa, Ya.

TITLE: Study of the Solubility of Indium Hydroxide¹ in Sodium Hydroxide Solutions

PERIODICAL: Zhurnal neorganicheskoy khimii, 1960, Vol. 5, No. 9, pp. 1996-1998

TEXT: The solubility of indium hydroxide in sodium hydroxide solutions with concentrations of from 1 to 17 mol/l was studied at 25°C. Indium hydroxide was prepared according to the method by N. A. Tananayev (Ref.6) and subsequently converted into the crystalline state according to the method by Fricke and Seitz (Ref. 7). The analysis results of crystalline indium hydroxide, dried at 120°C, corresponded to formula $In(OH)_3$. The solubility of indium hydroxide in solutions of sodium hydroxide at 25°C is given in Table 1, and Fig. 1 shows graphically the dependence of the solubility of indium hydroxide at 25°C on the concentration of sodium

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Study of the Solubility of Indium Hydroxide
in Sodium Hydroxide Solutions

S/078/60/005/009/029/040/KK
B017/B058

hydroxide. The maximum solubility of $\text{In}(\text{OH})_3$ in sodium hydroxide solutions (11.33 mol NaOH/l) amounts to 11.0 g/l. The solid phase consists of indium hydroxide at a soda lye concentration of up to 11.0 g/l. and of a hydrate of sodium hexahydroxo indate at a soda lye concentration above 11.0 g/l. The solubility of anorphous and crystalline indium hydroxide is the same. The authors mention E. A. Ostroumov, N. V. Aksel'rud, V. B. Spivakovskiy, E. N. Deychman, V. P. Chalyy and S. P. Rozhenko. D. Okhodnitski and Ya. Chizhniar participated in the study. There are 1 figure, 1 table, and 8 references: 6 Soviet, 1 French, and 1 German.

SUBMITTED: June 6, 1959

Card 2/2

S/078/60/005/012/015/016
B017/B064

AUTHORS: Ivanov-Emin, B. N., Nisel'son, L. A., Ivolgina, A. T.

TITLE: Study of the Solubility of Scandium Hydroxide in Sodium Hydroxide Solutions

PERIODICAL: Zhurnal neorganicheskoy khimii, 1960, Vol. 5, No. 12, pp. 2841-2842

TEXT: The solubility of scandium hydroxide in sodium hydroxide solutions containing 1 - 19 moles of NaOH/l was studied at 25°C. Maximum solubility of Sc(OH)₃ in an 11.7-mole solution of sodium hydroxide is 5.0 g/l. The existence of peaks on the solubility curve indicates the formation of sodium hydroxo scandiate. The solid phase up to the peak is crystalline scandium hydroxide, and the solid phase behind the peak is a hydrate of sodium hexahydroxo scandiate Na₃[Sc(OH)₆]·2H₂O. N. A. Tananayev is mentioned. There are 1 figure, 1 table, and 9 references: 4 Soviet, 2 British, 2 German, and 1 Czechoslovakian.

SUBMITTED: December 30, 1959
Card 1/1

8/080/60/033/008/005/013
A003/A001

AUTHORS: Nisel'son, L.A., Sokolova, T.D.

TITLE: An Investigation of the Kinetics of Interaction Between the Higher Oxides of Tantalum, Niobium, Titanium and Zirconium and Phosphorus Pentachloride _↙ _↙ _↙ _↙

PERIODICAL: Zhurnal prikladnoy khimii, 1960, Vol. 33, No. 8, pp. 1755-1765

TEXT: The kinetics of chlorination of higher oxides of tantalum, niobium, titanium and zirconium with liquid phosphorus pentachloride¹ was studied. The investigation was based on the assumptions 1) that chlorination in the condensed phase causes only a slight change in the concentration of the chlorinating agent; 2) that the experimental arrangement of the investigation is easy; 3) that chlorination products are obtained which can be used for the separation and purification of zirconium, tantalum and niobium by the rectification method. The results obtained in the experiments, presented in the form of a relation between the logarithm of oxide chlorination rate and the inverse absolute temperature of chlorination, obey in all cases the linear law. This proves that in the temperature interval studied (150-300°C) the chlorination processes take place in the kinetic region, i.e., the rate of the process is determined by the rate of the

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S/O80/60/033/008/005/013
A003/A001

An Investigation of the Kinetics of Interaction Between the Higher Oxides of Tantalum, Niobium, Titanium and Zirconium and Phosphorus Pentachloride

chemical interaction. Based on the tangents of the inclination angles of the curves obtained the activation energies of the process were calculated. The values obtained vary within the narrow limits of 25-33 kcal/mole and change little at the transition from one oxide to the other. For zirconium dioxide the activation energy is 11.0 kcal/mole, but the reproducibility of the results is low. The chlorination rate of oxides in relation to the weight unit (in g/g·min) depends mainly on the temperature of their preliminary calcination. The principal cause of the change in the oxide chlorination rate in this case is the change of their specific surface. The character of the modification of the oxides studied has only a slight effect on the chlorination rate. The chlorination rate of γ -Nb₂O₅ (calcinated at 800°C) differs from that of α -Nb₂O₅ by only 1.3 times, whereas for the same monoclinic modification of zirconium dioxide, but calcinated at 600 and 800°C the chlorination rates differ by a factor of 5.6. There are 6 figures, 1 table and 18 references: 12 Soviet, 2 American, 2 French, 1 English and 1 German.

SUBMITTED: December 21, 1959

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S/076/60/034/037/016/042/XX
B004/B0'8

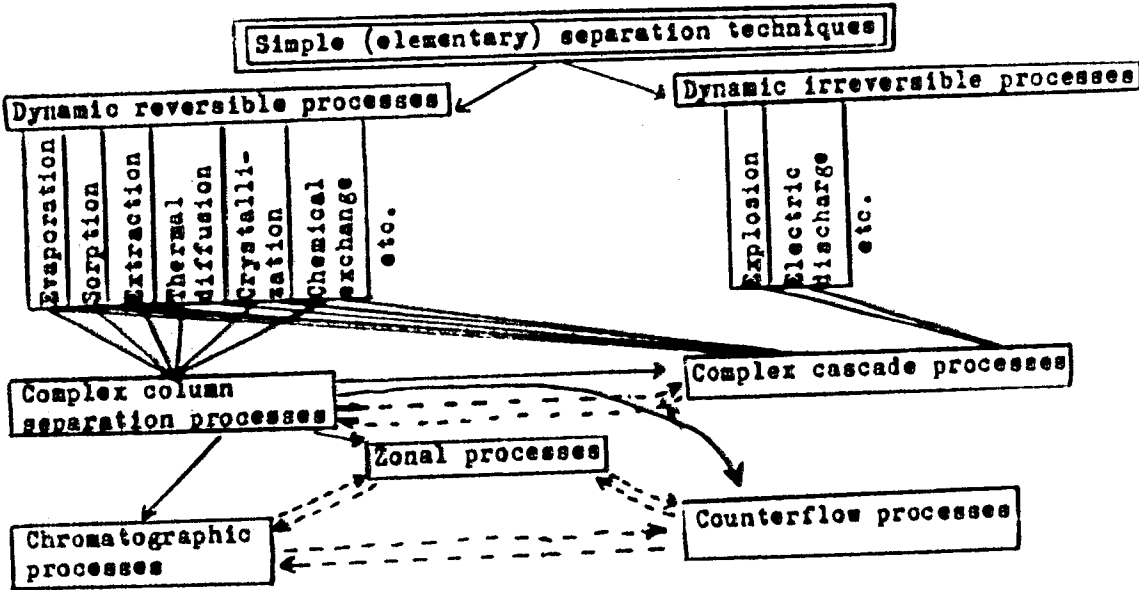
AUTHOR: Nisel'son, L. A.
TITLE: Classification of Separation Techniques
PERIODICAL: Zhurnal fizicheskoy khimii, 1960, Vol. 34, No. 7,
pp. 1460-1469

TEXT: This paper is an attempt to classify complex separation techniques according to the different dynamics of material flow of the components to be separated. The concepts of "dynamically reversible" and "dynamically irreversible" elementary processes are introduced. The material flow of three column processes is discussed in more detail, i.e., 1) counterflow; 2) zonal flow; and 3) chromatographic processes. These three processes are types of limiting cases. Multistage column processes may consist of combinations of these types. Transitions from one type to another are possible. A classification scheme is suggested: J

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Classification of Separation Techniques

S/076/60/034/007/016/042/XX
B004/B068



Card 2/3

Classification of Separation Techniques

S/076/60/034/007/016/042/XX
B004/B068

On the basis of this classification, new processes can be developed which have not been used by now. Comparison on the basis of some common parameter is suggested for determining the efficiency E : $E = G/\Pi \cdot (dN/d\tau)$. G is the quantity of the main component; Π is the parameter chosen; $dN/d\tau$ is the variation of the second component with time. There are 4 figures and 14 references: 7 Soviet, 3 US, 1 British, 2 German, and 1 Swiss.

ASSOCIATION: Moskovskiy institut tsvetnykh metallov i zolota im. K. I. Kalinina
(Moscow Institute of Nonferrous Metals and Gold imeni K. I. Kalinin)

SUBMITTED: September 15, 1958

Card 3/3

MISLISON, L.A.; IVANOV-SMIL, B.M.; LARIONOVA, L.Ye.

Crystals-liquid phase equilibria in binary systems formed by $ZrCl_4$,
 $HfCl_4$, $SnCl_4$, and $BiCl_3$. Zhur. neorg. khim. 6 no.1:186-191 '61. 4
(Zirconium chloride) (Hafnium chloride)
(Phase rule and equilibrium) (M.A. 14:2)

S/076/61/006/001/018/022
B121/B208AUTHORS: Nisel'son, L. A., Petrusovich, I. V.TITLE: Phase equilibria in the systems $\text{SiCl}_4 - \text{BCl}_3$ and $\text{SiBr}_4 - \text{BBr}_3$

PERIODICAL: Zhurnal neorganicheskoj khimii, v. 6, no. 3, 1961. 748-749

TEXT: The phase equilibria in the systems $\text{SiCl}_4 - \text{BCl}_3$ and $\text{SiBr}_4 - \text{BBr}_3$ were studied. The starting materials were purified by continuous boiling with copper filings and subsequent rectification. The systems were thermally analyzed by the heating curves recorded by Kurnakov's pyrometer. The melting-point diagrams of the systems studied are of the eutectic type. The heat of solution of SiCl_4 in BCl_3 was found to be 2.0 kcal/mole; the heat of solution of SiBr_4 in BBr_3 was 2.9 kcal/mole. A chemical reaction between the components in the systems $\text{SiCl}_4 - \text{BCl}_3$ and $\text{SiBr}_4 - \text{BBr}_3$ was not observed. The vapor pressure of the saturated BBr_3 vapors may be expressed by the equation

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S/078/61/006/003/022/022
B121/B208

AUTHORS: Vaks, S. A., Seryakov, G. V., Nisel'son, L. A.,
Sidorina, L. S.

TITLE: Liquid-vapor equilibrium in systems formed from the tetra-
chlorides of titanium, silicon, and carbon

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 6, no. 3, 1961, 756-759

TEXT: The equilibrium between liquid and vapor (at 760 mm Hg) in the
systems $TiCl_4 - SiCl_4$, $TiCl_4 - CCl_4$, and $CCl_4 - SiCl_4$ was studied
refractometrically at 20°C. The tetrachlorides had been purified by
distillation, and the titanium and silicon chlorides also chemically.
Data on the liquid-vapor equilibrium in the systems $TiCl_4 - SiCl_4$,
 $TiCl_4 - CCl_4$, and $CCl_4 - SiCl_4$ at 760 mm Hg are summarized in a table.
The refractive index in the systems $TiCl_4 - CCl_4$ and $TiCl_4 - SiCl_4$
was found to be a linear function of the composition. In the system

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Liquid-vapor equilibrium...

S/078/61/006/003/022/022
B121/B2 08

$TiCl_4 - SiCl_4$, a negative deviation from Raoult's law was found on the side of the lower-boiling component. The system $TiCl_4 - CCl_4$ is nearly ideal, while the system $CCl_4 - SiCl_4$ distinctly differs from the ideal state with respect to the course of the interface between liquid and vapor. There are 4 figures, 1 table, and 3 references: 1 Soviet-bloc and 2 non-Soviet-bloc. ✓

SUBMITTED: August 2, 1960

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Liquid-vapor equilibrium...
Card 3/3

S/078/61/006/003/022/022
B121/B208

① Content $TiCl_4 - SiCl_4$				② Content $TiCl_4 - OCl_2$				③ Content $OCl_2 - SiCl_4$			
t, °C	② Composition $SiCl_4$, mol. %		③ Structure $SiCl_4$ (1)	t, °C	② Composition OCl_2 , mol. %		③ Structure OCl_2 (1)	t, °C	② Composition $SiCl_4$, mol. %		③ Structure $SiCl_4$ (1)
	a) in liq. phase	b) in vap. phase			a) in liq. phase	b) in vap. phase			a) in liq. phase	b) in vap. phase	
57.2	100	100	—	78.5	100	100	—	57.2	100	100	—
57.9	87.86	99.85	6.78	77.0	97.97	99.815	5.4	58.1	92.5	95.3	1.64
59.7	84.18	99.22	7.57	76.5	98.9	97.88	5.2	60.0	78.3	85.3	1.61
60.9	87.84	98.09	7.15	83.8	77.7	85.03	5.5	61.6	67.0	78.0	1.60
61.9	85.0	98.0	8.7	88.4	88.5	92.0	5.9	63.1	60.6	72.1	1.68
63.8	85.2	97.16	8.5	93.7	52.4	85.2	5.2	64.2	54.0	66.5	1.70
66.1	73.15	96.18	9.3	102.4	37.0	74.5	5.0	67.2	36.7	50.5	1.75
69.1	66.4	84.78	9.25	112.0	23.0	58.5	4.9	68.7	27.6	41.0	1.82
78.0	49.5	81.8	10.8	125.2	10.1	36.2	5.1	71.2	19.3	30.0	1.79
88.9	30.4	83.2	11.4	131.8	3.10	12.8	4.6	72.8	12.2	20.2	1.82
105.2	17.0	86.5	9.7	136.5	0	0	—	74.4	6.6	11.5	1.83
116.5	8.7	46.4	8.1					76.5	0	0	—
123.4	5.5	33.2	8.8								
129.0	2.50	18.5	8.8								
132.5	0.81	8.0	9.4								
134.4	0.37	3.8	8.9								
135.6	0.19	1.58	9.3								
136.4	0	0	—								

Legend to the Tables:

- 1) system;
- 2) content, mol. %;
- a) in liquid phase;
- b) in vapor phase;
- 3) relative fugacity.

IVANOV-EMIN, B.N.; NISEL'SON, L.A.; RABCVIK, Ya, E.; LARIONOVA, L.Ye.

Complex compounds of gallium halides with o-phenanthroline. Zhur.
neorg.khim. 6 no.5:1142-1146 May '61. (MIRA 14:4)

(Gallium compounds) (Phenanthroline)

WISL'SON, L.A.

Density of $ZrCl_4$ and $HfCl_4$ in the vapor and liquid state. Zhur.
neorg.khim. 6 no.5:1242-1244 My '61. (MIRA 14:4)

(Zirconium chloride) (Hafnium chloride)

IVANOV-EMIYE, B.N.; NISEL'SON, L.A.; IVOLGINA, A.T.

Solubility of yttrium hydroxide in sodium hydroxide solutions.
Zhur.neorg.khim. 6 no.6:1483-1484 Je '61. (MIRA 14:11)
(Yttrium oxide) (Sodium hydroxide)

NESEL'SON, L.A.; SOKOLOVA, T.D.

Liquid-vapor phase equilibrium in systems formed by $TiCl_4$,
 $VOCl_3$, and $POCl_3$. Zhur. neorg. khim. 6 no.7:1645-1651 J1
'61. (MIRA 14:7)

(Systems (Chemistry))

S/080/61/034/011/001/020
D201/D301

AUTHORS: Ivanov-Emin, B.N., Nisel'son, L.A., and Petrushevich, L.V.

TITLE: The synthesis and purification of boron bromide and iodide

PERIODICAL: Zhurnal prikladnoy khimii, v. 34, no. 11, 1961, 2378 - 2384

TEXT: The authors carried out experiments for preparing boron bromide and iodide directly from the elements. As starting materials pure bromine and iodine and samples of amorphous boron containing 87 - 99.5 % B were used. The halogenations were performed in vertical quartz reactors, 3 - 5 cm in diameter. Boron was bricketed with a sugar solution in methanol and the hologenation process was carried out in a stream of argon at 640 - 1050°C for 3 - 6 hours. During halogenation fractions of the halodes were analyzed for free Br₂ and I₂ contents. For comparison purposes, one bromation reaction was performed in the reactor lying almost horizontally. All ex-
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The synthesis and purification ...

S/080/61/034/011/01/020
D201/D301

perimental details are given in full. The yields were markedly higher in vertical reactors than in the horizontal ones. the optimum temperature being 850 - 900°C for both halogenation reactions; the bromination yield was 94.5 - 99.5 % B Br₃. The iodination process was quite similar to that of bromination, but with lower yields (63.5 - 50 %). Purification of the crude halides, as well as of technical B Cl₃, was carried out by distillation in a rectifying column of a plate-sieve type, with 25 real plates. The rectification of technical BCl₃ and that of the crude BBr₃ was easily carried out: after the first distillation BCl₃ was practically free from SiCl₄ and COCl₂, present in the initial product, and BBr₃ was practically free from SiBr₄ and other volatile components; a pure colorless product was obtained by a second distillation in presence of copper filings. Purification of the crude iodide was not so easy owing to the high iodide content in the product. This was overcome by converting the free I₂ to SnI₄, with metallic tin. The authors were able, by this method, to obtain from 5.2 kg of the iodination product, containing about 25 % of SnI₄, 0.4 % SiI₄ and some other impurities, 2.05 kg of practically pure BI₃, after one fractional

Card 2/3

The synthesis and purification ...

S/080/61/034/011/001/020
D201/D301

distillation. There are 3 figures and 4 tables and 11 references:
6 Soviet-bloc and 5 non-Soviet-bloc. The references to the English-
language publications read as follows: E. Gamble, P. Gilmont, J.
Stoff, J. Am. Chem. Soc., 62, 1257, 1940; W. Schumb, E. Gamble, M.
Banus, J. Am. Chem. Soc., 71, 3225, 1949.

SUBMITTED: December 12, 1960

Card 3/5

S/828/62/000/000/015/017
E071/E135

AUTHOR: Nisel'son, L.A.

TITLE: Separation and purification of halogenides of zirconium and hafnium, niobium and tantalum by methods based on differences in the volatility

SOURCE: Razdeleniye blizkikh po svoystvam redkikh metallov. Mezhvuz. konfer. po metodam razdel. blizkikh po svoyst. red. metallov. Moscow, Metallurgizuat, 1962, 168-185.

TEXT: The possibilities of separation of the above elements by rectification are discussed and supported by experimental data obtained on a laboratory apparatus. The following processes were examined: separation and purification of tantalum and niobium by rectification of products of interaction of their pentachlorides with phosphorus oxychloride; rectification of pentachlorides of tantalum and niobium; separation of zirconium and hafnium by rectification of products of interaction of their tetrachlorides with phosphorus oxychloride; separation of zirconium and hafnium by extractive rectification of their tetrachlorides [Abstractor's note: this process is discussed but no experimental work is

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Separation and purification of ...

S/828/62/000/000/015/017
E071/E135

reported] ; separation of zirconium and hafnium by rectification of their tetrachlorides under pressure. The latter process is of particular interest for industrial application but it presents difficult problems. These could be solved by designing special apparatus applying reliable temperature monitoring, salts as heat carriers, voltage stabilization and automation of the process with a view to continuous operation.

There are 3 figures and 4 tables.

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S/828/62/000/000/016/017
E071/E135

AUTHORS: Zelikman, A.N., Kreyn, O.Ye., Nisel'son, L.A.,
Gorovits, N.N., and Ivanova, Z.I.

TITLE: Separation of tungsten and molybdenum by utilizing the
difference in volatility of their chlorides and
oxychlorides

SOURCE: Razdeleniye blizkikh po svoystvan redkikh metallov.
Mezhvuz. konfer. po metodam razdel. blizkikh po svoyst.
red. metallov. Moscow, Metallurgizdat, 1962, 186-197.

TEXT: A method of separating tungsten from molybdenum, based
on evaporation of MoO_2Cl_2 on heating of molybdenum trichloride
with sodium chloride to a temperature of 600-700 °C, was studied.
With contents of 0.01 to 0.16 and 1.035% W in the starting
molybdenum trioxide the purified product contained less than
(6 to 9) $\times 10^{-4}$ and 1.5×10^{-3} % W respectively. It was established
that it is possible to separate tungsten and molybdenum by
rectification of their higher chlorides, WCl_6 and $MoCl_5$
(rectification column data: diameter 30 mm, height 600 mm,
15 sieve plates, with 45 holes of 1 mm diameter).
Card 1/2

Separation of tungsten and molybdenum... S/828/62/000/000/016/017
E071/E135

From tungsten sexquichloride containing about 5% $KoCl_5$, and from molybdenum pentachloride containing about 5% WCl_6 , purified chlorides containing below 0.01% of admixture of molybdenum or tungsten respectively with yields of the main fractions of 70-80% were obtained.

There are 6 figures and 7 tables.

Card 2/2

33279

S/078/62/007/002/006/019

B119/B110

5.4210 1087

AUTHORS: Nisel'son, L. A., Voytovich, B. A.TITLE: The SiCl_4 - POCl_3 - BCl_3 system

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 7, no. 2, 1962, 360 - 363

TEXT: The equilibria between crystalline and liquid phases and between liquid and vapor phases of the systems POCl_3 - BCl_3 and SiCl_4 - POCl_3 - BCl_3 were studied. The methods are described by the first author in Zh. neorg. khimii, 5, 1139 (1960); 3, 2150 (1958); and I. A. Shcheka et al., 1, 964 (1956). This study is of interest for obtaining purest Si. Results: BCl_3 and POCl_3 form in solid state the compound $\text{BCl}_3 \cdot \text{POCl}_3$ which dissociates almost completely on melting (melting point 83.8°C). (The equation of A. B. Mlodzyevskiy and O. A. Yesin can be used for calculating the dissociation constant immediately above the melting point.) The heat of solution of $\text{BCl}_3 \cdot \text{POCl}_3$ in SiCl_4 is 4.6 kcal/mole. When dissolved in SiCl_4 , the compound is totally dissociated (in BCl_3 and POCl_3). The

Card 1/2

The SiCl_4 - POCl_3 - BCl_3 system

33279
S/078/62/007/002/006/019
B119/B110

simultaneous presence of small BCl_3 and POCl_3 quantities in SiCl_4 has no influence on their relative volatility as to SiCl_4 . There are 4 figures, 2 tables, and 9 references: 7 Soviet and 2 non-Soviet. The reference to the English-language publication reads as follows: A. B. Burg, M. R. Poss. J. Amer. Chem. Soc., 65, 1637 (1943). X

ASSOCIATION: Gosudarstvennyy institut redkikh metallov (State Institute of Rare Metals)

SUBMITTED: December 24, 1960

Card 2/2

IVANOV-EMIN, B.N.; NISEL'SON, L.A.; LARIONOVA, L.Ie.

Properties of solutions of alkali metal gallates. Zhur.neorg.khim.
7 n.3:522-526 Nr '62. (MIRA 15:3)
(Gallic hydroxide)

31868
S/078/62/007/005/015/019
B110/B158

21,2500

AUTHOR: Nisel'son, L. A.

TITLE: Constitution diagrams of binary systems formed by zirconium and hafnium halides

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 7, no. 3, 1962, 693 - 694

TEXT: The crystal - liquid phase equilibrium was investigated in binary systems of zirconium and hafnium tetrachlorides, tetrabromides, and tetraiodides. The halides were produced from Zr (0.05% Hf), Hf (0.3% Zr), Cl, Br, and I. The binary systems $ZrCl_4$ - $HfCl_4$, $ZrBr_4$ - $HfBr_4$, ZrI_4 - HfI_4 exhibit unlimited solubility in the liquid and solid states. Their constitution diagrams show an almost straight-line course for the liquidus curve with the solidus almost beside it. This is because the Zr and Hf atomic radii are almost equal, the tetrahalide crystal lattices are identical, and their melting points are very close. The systems ZrI_4 - $ZrCl_4$ and $ZrBr_4$ - $ZrCl_4$ constitute a continuous series of solid solutions with a minimum. ZrI_4 - $ZrCl_4$ has a lower minimum (~ 50 mole% of $ZrCl_4$).

Card 1/2

Constitution diagrams of binary...

S/078/62/007/003/015/019
B110/B138

~3680C) due to the greater difference between the atomic radii of I and Cl than between Br and Cl. There are 2 figures, 1 table, and 3 references: 2 Soviet and 1 non-Soviet.

SUBMITTED: October 4, 1960

✓

Card 2/2

S/078/62/007/005/004/014
B101/B110

AUTHORS: Nisel'son, L. A., Teslitskaya, M. V., Shvedova, T. A.
TITLE: Synthesis of zirconium(IV) iodide and hafnium(IV) iodide by halogen exchange
PERIODICAL: Zhurnal neorganicheskoy khimii, v. 7, no. 5, 1962, 971 - 974

TEXT: The following reactions are discussed for preparing pure $Zr(Hf)I_4$:
(A) $ZrCl_4 + 4NaI \rightleftharpoons ZrI_4 + 4NaCl$. The initial mixture was molten in an evacuated ampoule (650-700°C), and the ampoule heated on one side (initially 400°C, finally 650-700°C) to effect sublimation of the substance into the colder part. The sublimate contained 85.5% by weight of ZrI_4 and 14.5% by weight of $ZrCl_4$. Complete separation of the chloride from the iodide was not attained. (B) $ZrCl_4 + SiI_4 \rightleftharpoons ZrI_4 + SiCl_4$ (at 250-320°C) also yielded only 78% substitution of chlorine by iodine. (C) $3Zr(Hf)Cl_4$

Card 1/3

Synthesis of zirconium(IV) iodide ...

S/078/62/007/005/004/014
B101/B110

+ $2\text{Al}_2\text{I}_6 \rightleftharpoons 3\text{Zr}(\text{Hf})\text{I}_4 + 2\text{Al}_2\text{Cl}_6$ (at $300-365^\circ\text{C}$) produced a 100% yield of ZrI_4 or HfI_4 , respectively, containing 0.1-0.05% Al. The reaction with Al_2Br_6 yielded only 80-85% substitution. (D) $\text{ZrCl}_4 + 4\text{HI} \rightleftharpoons \text{ZrI}_4 + 4\text{HCl}$. Because of the high vapor pressure of the resulting HCl, work was carried out at atmospheric pressure, and the HI in Ar as carrier gas was conducted over ZrCl_4 heated to 370°C . 81.5-82% substitution was attained. (E) Since ZrI_4 and ZrCl_4 form no stable compounds with each other and have different volatilities, their separation was attempted by distillation. A mixture of 29.4% by weight of ZrCl_4 and 70.6% by weight of ZrI_4 was heated to $400-420^\circ\text{C}$, finally to $500-520^\circ\text{C}$. The ampoule was cut into zones, and the condensates of the individual zones were analyzed. Fraction I consisted of ZrCl_4 with only 0.1% ZrI_4 ; fraction V consisted of 95% ZrI_4 ; and the distillation residue of 100% Al-free ZrI_4 , while the initial mixture contained 0.1% Al. Besides this separation, the impurity was also removed.

Card 2/3

IVANOV-EMIN, B.N.; NISEL'SON, L.A.; GVOZDEVA, N.I.

Solubility of gallium hydroxide in sodium hydroxide and potassium
hydroxide solutions at 25°C. Zhur.neorg.khim. 7 no.5:1150-1153
My '62. (MIRA 15:7)

(Gallium hydroxide) (Alkalies) (Solubility)

S/078/62/007/012/002/022
B144/B180

AUTHORS: Nisel'son, L. A., Sokolova, T. D.

TITLE: Orthobaric densities and critical parameters of zirconium
(IV) and hafnium (IV) halides

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 7, no. 12, 1962, 2655-2660

TEXT: From the orthobaric densities determined in high-purity Zr and Hf tetraiodides, tetrabromides and tetrachlorides, the critical values of these compounds were calculated. The vapor and liquid densities were determined in a range from about 360 to 690°C in a transparent oven containing a nitrite-nitrate bath for temperatures below 500°C or a nickel block for higher temperatures. The sample enclosed in a sealed ampoule or a pycnometer was placed in the bath or block. The values found for $ZrCl_4$ and $HfCl_4$ were somewhat higher and had a flatter maximum than those published earlier (Zh. neorgan. khimii, 6, 1242 (1961)). The critical temperature was derived from the maximum of the orthobaric density. The critical density was found according to the Cailletet-Mathias law; for
Card 1/2

37547

S/080/62/035/005/002/015
D204/D307

5.2410

AUTHORS: Eisel'son, L. A., Petrushevich, I. V., Shamray, F. I.
and Fedorov, T. F.

TITLE: Preparation of elemental boron by reduction of its
halides with hydrogen

PERIODICAL: Zhurnal prikladnoy khimii, v. 35, 1962, 984-989

TEXT: The present work was carried out to supplement existing data on the preparation of elemental B. Purified BCl_3 , BBr_3 , BI_3 and H_2 were used under anhydrous conditions. The reactor consisted of a quartz tube enclosing a pair of Mo electrodes connected by a Ta wire $100 \times 8 \times 0.10 \text{ mm}$, on which the B was deposited. Temperatures were varied from 800 to 1400°C and the molar $(\text{BX}_3):(\text{H}_2)$ ratios, (n), were made 1:3-25. The interactions took place over 1.7 - 3 hours. The rate of B deposition increased rapidly with temperature and tended to be higher for lower n. For BBr_3/H_2 mixtures the

Card 1/2

Preparation of elemental ...

S/080/62/035/005/002/015
D204/D307

yields of B increased linearly from ~3% at 800°C to ~50% at 1300°C, almost independently of n. For a given temperature the rate of B deposition increased in the order $BCl_3 < BBr_3 < BI_3$. Between 800 and 900°C brown, friable deposits of amorphous B were obtained for all n studied, from BBr_3/H_2 mixtures. At higher temperatures and n, black, "graphite" B was produced, and "metallic" crystalline boron formed at and above 1200°C with 3 or less moles H_2 /mole BBr_3 . Similar regions of formation of each form of B are believed to exist for BCl_3-H_2 and BI_3-H_2 systems. There are 6 figures and 1 table.

SUBMITTED: January 25, 1961

Card 2/2

S/080/62/035/003/002/024
D258/D302

AUTHORS: Seryakov, G. V. and Nisel'son, L. A.

TITLE: The relationship between relative volatility and mutual solubility

PERIODICAL: Zhurnal prikladnoy khimii, v. 35, no. 3, 1962, 482-486

TEXT: In continuation of earlier work the authors studied the influence of limited mutual solubility on the relative volatility of components in binary systems. It was assumed in the case of a binary system of limited solubility that the vapor pressures of the solvent and solute in each region obeyed Raoult's and Henry's laws respectively and the relationship between the mole fractions, y and x , of component A in the gaseous and liquid phases respectively, was thus given for the A-rich region by

$$y = \frac{P_A \cdot x}{P_A \cdot x + K_B(1 - x)} \quad (2)$$

Card 1/3

The relationship between ...

S/080/62/035/003/002/024
D258/D302

where P_A was the vapor pressure of pure liquid A and K_B Henry's constant for B. K_B was expressed as $P_B = (1 - X_2)/(1 - X_1)$, where X_1 and X_2 were the mole-fractions of A in liquid phases rich in either A or B respectively. The relative volatility for the A-rich region was

$$\alpha_{A/B} = \frac{y(1-x)}{x(1-y)} = \frac{P_A}{P_B} \cdot \frac{x_1}{x_2} \quad (5)$$

The limiting value of (5), as $X_2 \rightarrow 0$, was:

$$\alpha_{A/B} = \alpha_{A/B \text{ ideal}} \cdot X_1 [B]$$

Card 2/3

The relationship between ...

S/080/62/035/003/002/024
D258/D302

where $X_1^{[B]}$ was the mole fraction of B in a saturated solution, rich in A. This correction to Raoult's law was checked experimentally with solutions of Fe_2O_3 in $TiCl_4$ and $SiCl_4$; Al_2Cl_6 in $SiCl_4$; $SbCl_3$ in $SiCl_4$; and Mg in Na. The experimental data were in agreement with the calculated values. There are 1 table and 4 Soviet-bloc references.

SUBMITTED: June 26, 1960

Card 3/3

NISEL'SON, L.A.; PETRUSEVICH, I.V.; SHARAY, F.I.; FEDOROV, T.F.

Preparation of elemental boron by hydrogen reduction of its
halide. Zhur.prikl.khim. 35 no.5:984-989 My '62. (MIRA 15:5)
(Boron) (Boron halides) (Hydrogen)

S/030/62/035/007/004/013
D267/D307

AUTHORS: Zelikman, A.N., Kreyn, O.Ye., Nisel'son, L.A. and Ivanova, E.I.

TITLE: Separation of tungsten from molybdenum by the rectification of their chlorides

PERIODICAL: Zhurnal prikladnoy khimii, v. 35, no. 7, 1962, 1467-1472

TEXT: $WOCl_6$ and $MoCl_5$ were obtained from pure metals by chlorination at 600-750°C, distilled in an argon atmosphere to separate the oxochlorides, after which $WOCl_6$ with about 5% $MoCl_5$ or vice versa were rectified on a plate column. It was found that the impurity content of the purified chloride is less than 0.015%, and that the yield of the rectified chloride is 70-80% of theoretical. There are 5 figures and 3 tables.

SUBMITTED: June 21, 1961

Card 1/1

NISEL'SON, L.A.; VIGDOROVICH, V.N.; SERVAKOV, . V.

Interphase distribution of components in the low concentration region. Zhur. fis. khim. 36 no.4:697-702 Ap '62. (MIRA 15:6)

1. Gosudarstvennyy nauchno-issledovatel'skiy i proyektnyy institut redkometallicheckoy promyshlennosti i Institut tsvetnykh metallov imeni N.I. Kalina.

(Systems (Chemistry)) (Phase rule and equilibrium)

L 17126-63

EWP(q)/EWT(π)/EDS APFTC RDW/JD/AR

ACQUISITION NR: AP3000905

s/0279/67/000/002/0079/0085

AUTHORS: Nigel'son, L. A.; Fustil'nik, A. I.; Sashnikova, L.A. (Moscow)61
59TITLE: Purifying selenium from tellurium by rectificationSOURCE: M' SSSR. Izv. otd. tekhn. nauk. Metallurgiya i gornaya delo, no. 2, 1963, 79-85

TOPIC TAGS: rectification, purification, Se, Te, density, viscosity, surface tension

ABSTRACT: The authors made ⁶their experiment because the common technique of obtaining Se (by distillation) is ineffective in eliminating certain elements that have comparable volatility (especially Te, Sb, and Sn). Because of complications in construction if rectification were carried on in a vacuum and because such properties as viscosity and surface tension are thus altered deleteriously, it appeared best to rectify Se at ordinary atmospheric pressure. The setup is illustrated in Fig. 1 (see Enclosure 1). The internal diameter of the column is 30-32 mm. The sieve plate has 40 openings 0.8 mm in diameter, formed ultrasonically. The distance between plates is 30-32 mm, and 10 plates are used in the column. To prevent congealment of the Se, the head of the apparatus is equipped with an electrical

Card 1/3

L 17126-63

ACCESSION NR: AF3000905

2

heating element. The thermally insulated jacket of the column, with its electrical heater, is made of glass tubing with asbestos insulation, and the jacket has a transparent window for observation. The temperature was measured in tests with an accuracy of 0.50 by a Chromel-Alumel thermocouple and a semi-automatic E2/11 potentiometer. In testing the equipment, rectification of Se suffered from the difficulty of maintaining normal conditions, resulting from unusual (impulsive) boiling of Se and from the very narrow range of operating flow rates into the column. The degree of purification obtained in the experiments proved to be substantially less than computed values indicated they should be. The authors conclude that this is due partly to the problem of maintaining steady conditions and partly to imperfections in the design of the column head. They are convinced the rectification method has great promise for Se. Orig. art. has 4 figures and 6 tables.

ASSOCIATION: none

SUBMITTED: 06Aug62

DATE ACQ: 12Jun63

DECL: 01

SUB CODE: 42

NO REF SOV: 014

OTHER: 008

Card 2/12

L 18524-63

EPP(q)-2/EWP(q)/EWT(m)/RDS/ES(m)-2 APFIC/ASD/SSD Pt-L/Pu-L

ACCESSION NR: AP3002389

WW/JD/JG

8/0279/63/000/003/0110/0110

AUTHORS: Nicol'son, L. A.; Pustil'nik, A. I. (Moscow)TITLE: Density and viscosity of liquid niobium and tantalum pentachlorides 72SOURCE: M SSSR. Izv. Otd. tekhnicheskikh nauk. Metallurgiya i gornoye delo,
no. 3, 1963, 110 27

TOPIC TAGS: niobium pentachloride, tantalum pentachloride

ABSTRACT: Density (ρ) and viscosity (η) of liquid NbCl_5 and TaCl_5 have been determined at the temperature interval of 300-320°C. Each substance contained no more than 0.02% of impurities. Densities were determined in sealed quartz pycnometers, and temperatures were measured with both standard thermometers and thermocouples (temperature accuracy was ± 0.10). The results were corrected for the thermal expansion of quartz and for the weights of vapors. Relative error in density was $5 \times 10^{-2}\%$, in viscosity it was 0.2%. Viscosities were determined in a modified Martin viscosimeter. Data were processed by the method of least squares. It was established that:

$$\rho_{\text{NbCl}_5} = 2.0737 - 3.115 \cdot 10^{-5} \cdot \Delta t + 3.58 \cdot 10^{-8} \cdot \Delta t^2$$

$$\rho_{\text{TaCl}_5} = 2.0840 - 4.100 \cdot 10^{-5} \cdot \Delta t$$

Card 1/2

L 18524-63

ACCESSION NR: AP3002309

$$\eta_{\text{InCl}} = 0.921 - 1.325 \cdot 10^{-3} \cdot \Delta T + 1.120 \cdot 10^{-5} \cdot \Delta T^2 - 4.30 \cdot 10^{-7} \cdot \Delta T^3$$

$$\eta_{\text{TeCl}} = 1.003 - 1.687 \cdot 10^{-3} \cdot \Delta T + 1.8 \cdot 10^{-5} \cdot \Delta T^2 - 8.455 \cdot 10^{-7} \cdot \Delta T^3$$

Orig. art. has: 4 formulas.

ASSOCIATION: none

SUBMITTED: 31Jen63

DATE ACQ: 12Jul63

EXCL: 00

SUB CODE: CH

NO REF SOV: 004

OTHER: 000

Card 2/2

1/078/62/008/004/003/013
A059/A126

AUTHORS: Pugachevich, P.P., Nisel'son, L.A., Sokolova, T.D., Anurov, N.S.

TITLE: Density, viscosity, and surface tension of carbon tetrachloride and tin tetrachloride

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 8, no. 4, 1963, 791 - 796

TEXT: The density of CCl_4 and SnCl_4 was measured in a sealed quartz pycnometer (Fig. 1) with a volume of about 20 cm^3 and a capillary diameter of about 2 mm. The volume expansion of quartz was taken as $1.5 \cdot 10^{-6}$ in the calculations. The correction for the vapors in the free volume of the capillary was calculated from the ideal-gas equation where the saturated-vapor pressure was determined from the equations:

$$\text{CCl}_4 \dots \log p = \frac{-2400}{T} - 5.3 \log T + 23.6 . \quad (1)$$

and

$$\text{SnCl}_4 \dots \log p = \frac{-1022}{T} + 7.865 . \quad (2)$$

Card 1/5

3/078/63/008/004/003/013

AC59/1126

Density, viscosity, and surface tension of

The relative error can be about $5 \cdot 10^{-2}\%$. The viscosity was measured with the somewhat modified Martin viscometer made of molybdenum glass. The capillary diameter was selected between 0.3 and 0.5 mm, and the length of tube 4 was 180 mm. The viscosity was calculated from the equation:

$$\eta = c (\rho_l - \rho_v) \tau, \quad (3)$$

where c is the constant of the apparatus, ρ_l and ρ_v is the density of the liquid and the vapor, respectively, at a given temperature, and τ is the time of flow. The relative error of the viscosity determination is not in excess of 0.2%. The surface tension was measured with the setup shown in Figure 3, and calculated from the equation:

$$\sigma = \frac{1}{2} g (\rho_l - \rho_v) r h_1 \left[1 - \frac{2}{3} \frac{r}{h_1} - \frac{1}{3} \frac{r^2}{h_1^2} \right], \quad (4)$$

where g is acceleration due to gravity, r the inner radius of tube 6 at the top (in this case, $r = 0.010$ cm). The relative error in no case exceeded 0.2%. Equations relating density, viscosity, and surface tension of CCl_4 and SnCl_4 to temperature found by the least-square methods were: $\rho = 1.6287 - 0.001763 t -$

Card 2/5

S/078/63/008/004/003/013
A059/A126

Density, viscosity, and surface tension of

- $0.00000209 t^2$ (for CCl_4) and $\rho = 2.2789 - 0.0025437 t - 0.00000081 t^2$ (for SnCl_4); $\eta = 1.3418 - 0.022493 t + 0.0002222 t^2 - 0.000000946 t^3$ (for CCl_4) and $\eta = 1.0917 - 0.01241 t + 0.00007712 t^2 - 0.000000193 t^3$ (for SnCl_4); and $\sigma = 29.21 + 0.1259 t$ (for CCl_4) and $\sigma = 29.92 - 0.1134 t$ (for SnCl_4). There are 5 figures and 6 tables.

ASSOCIATION: Institut obshchey i neorganicheskoy khimii Akademii nauk SSSR (Institute of General and Inorganic Chemistry of the Academy of Sciences, USSR), Gosudarstvennyy nauchno-issledovatel'skiy i proyektiruyemyy institut redkometallicheskoy promyshlennosti (State Design and Planning Scientific Research Institute of the Rare Metal Industry)

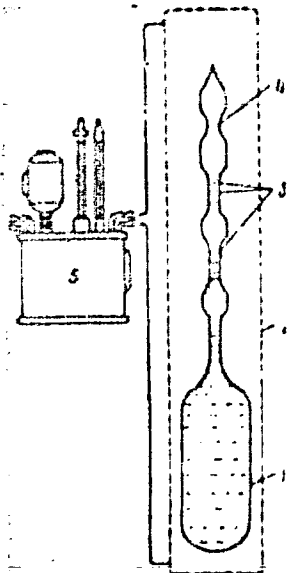
SUBMITTED: August 24, 1962

Card 3/5

Density, viscosity, and surface tension of

3/078/63/008/004/003/013
A059/A126

Figure 1: Pycnometer for the determination of the density of chlorides: 1 - pycnometer; 2 - constant temperature jacket; 3 - marks; 4 - place of opening of the pycnometer; 5 - Heeppler thermostat.

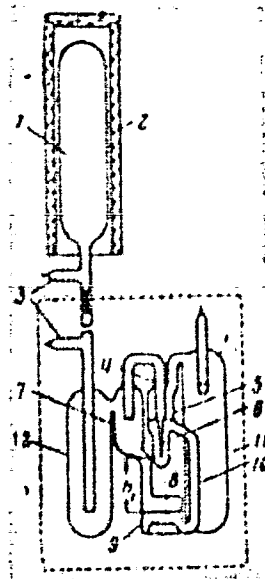


Card 4/5

S/073/63/008/004/003/013
A059/A126

Density, viscosity, and surface tension of ...

Figure 3: Improved gas setup for the determination of the surface tension: 1 - gas cylinder; 2 - electric furnace; 3 - tube of the apparatus for liquid and gas filling; 4, 8, 10 - connecting pipe; 5 - container for tube calibration; 6 - calibrated tube; 7 - intermediate cylinder; 9 - pressure-gauge cylinder; 11 - cushioning cylinder; 12 - trap.



Card 5/5

IVANOV-EMIN, E.N.; NISEL'SON, L.A.; SOKOLOVA, T.D.

Reactions of scandium chloride with ethylenediamine. Zhur.
neorg. khim. 8 no.6:1381-1383 Je '63. (MIRA 16:6)

(Scandium chloride)
(Ethylenediamine)

NISEL'SON, L. A.

TITLE: Seminar on refractory metals, compounds, and alloys (Izv, April 1963).

SOURCE: Atomnaya energiya, v. 15, no. 3, 1963, 266-267.

ACCESSION NR: AP3008085

Ye. I. Yelagina, K. Kh. Abrikosov. Synthesis and investigation of rhenium silicide.

G. P. Shveykin and others. Kinetics of niobium oxycarbide decomposition in vacuum, interaction of niobium and carbon monoxide, etc., in connection with the development of the carbothermal method of extraction of niobium from its oxides.

L. A. Nisel'son and others. Obtaining niobium, tantalum, and their alloys by reduction of gaseous chlorides with hydrogen on a heated surface.

G. V. Samsonov, S. N. L'vov, V. N. Paderno. Obtaining ZrC, HfC, NbC, and TaC solid solutions by hot compacting of mixtures of oxides with carbon.

V. F. Funke, V. I. Pshenichnyy. Study of conditions of obtaining TiC, ZrC, and VC from oxides.

V. N. Bondarev. Investigation of synthesis of transition-metal

Card 4/11

POLYAKOV, Ya.M.; NISEL'SON, L.A.; KRESTOVNIKOV, A.N.

Process for producing tantalum and niobium by the reduction of their
pentachlorides with hydrogen (reduction of $TaCl_5$). Zhur.prikl.khim.
36 no.1:25-33 Ja '63. (MIRA 16:5)
(Tantalum) (Niobium)

ACCESSION NR: AP4019808

S/0279/64/000/001/0067/0077

AUTHOR: Meyerson, G. A. (Moscow); Nisels' son, L. A. (Moscow); Chou, Ch'u-Ming (Moscow)

TITLE: Production of silicon carbide and its alloys with boron by reduction from the vapor phase over a heated surface

SOURCE: AN SSSR. Izv. Metallurgiya i gornoye delo, no. 1, 1964, 67-77

TOPIC TAGS: silicon carbide, silicon carbide production, silicon carbide alloy, boron containing alloy, boron containing silicon carbide alloy, vapor phase reduction, silicon tetrachloride reduction, silicon carbide crystallization

ABSTRACT: The authors discuss the existing literature on silicon carbide production, the growth of large, single, high-purity crystals, the α and β phases, the production of SiC from a vapor-gas mixture of SiCl_4 , toluene and hydrogen, etc. On calculating the changes in free energy for the formation of SiC from a mixture of SiCl_4 with some carbon-containing products in the presence and absence of hydrogen, they found that a substantial reduction of the isobaric potential accompanies the process when hydrogen is present; they conclude from their thermodynamic analysis that the initial materials must be carefully purified when high purity SiC is produced by deposition from a vapor-gas mixture over a heated surface. Their

Card 17

ACCESSION NR: AP4019808

experiments, carried out to study the physical and chemical conditions of deposition, employed a core of preliminarily carbided tantalum wire on which pure SiCl_4 was deposited in a reactor (illustrated) from carefully purified SiCl_4 . In the presence of electrolytically purified H_2 and toluene. Core temperature varied from 1400 to 2000C, the Si:C ratio in the initial vapor-gas mixture from 1 to 6, the rate of vapor-gas flow (normal atmospheric pressure, 25C) from 0.5 to 1.9 liters/min, and the amount of H_2 from 7 to 11 times the stoichiometric proportion. Pure SiC depositing at 1600-1800C represented the β phase; the optimal Si:C ratio was found to be 3:1, the excess Si being required to prevent deposition of free graphite. Other experiments concerned alloying SiC with B (1600-1650C, vapor-gas flow about 1.0 liter/min at 25C, hydrogen excess 11 times the stoichiometric proportion) by combined deposition from SiCl_4 , C_2H_6 and BBr_3 . The Si:C ratio in the initial mixture was 3:1 and constant, the amount of B varied up to 9% in relation to SiC. The B content in the deposit corresponded to the B:C ratio in the vapor-gas phase, and the microhardness of SiC was increased by addition of B. Various analyses of deposits with up to 8.5% B by weight indicated retention of a uniform solid-solution pattern of B substitution in the β -phase of SiC. An analysis of the kinetics of the SiC deposition process showed that the rate peaks at 1700-1800C, while its dependence on reagent concentration peaks around 2.5 g/liter (an 8-11 times excess of H_2 is optimal); the rate increases with rising velocity

Card 2/3

ACCESSION NR: AP4019808

of the vapor-gas flow and reaches about $0.4 \text{ g/cm}^2\text{-hr}$ at 1600C , 1.8 liters/min (25C) for a tubular reactor with a diameter of 50 mm . The yield drops to 65% in relation to C. Introduction of B slows the rate of deposition to some extent. Orig. art. has: 2 tables, 7 graphs and 10 formulas.

ASSOCIATION: none

SUBMITTED: 06Aug63

DATE ACQ: 31Mar64,

ENCL: 00

SUB CODE: NL, CH

NO REF SOV: 010

OTHER: 018

Card 1/3

ACCESSION NR: AP4017567

S/0149/64/000/001/0131/0138

AUTHOR: Polyakov, Ya. M.; Masl'son, L. A.; Krestovnikov, A. N.

TITLE: Preparation of niobium and tantalum from the vapor phase

SOURCE: IVUZ. Tsvetnaya metallurgiya, no. 1, 1964, 131-138

TOPIC TAGS: niobium, tantalum, niobium production, tantalum production, niobium coating, tantalum coating, vapor phase plating, niobium alloy, tantalum alloy

ABSTRACT: The following conclusions resulted from a review of Soviet and Western work on the preparation of niobium and tantalum from the vapor phase. The production of niobium and tantalum coatings, deposited from the vapor phase onto steel, iron, copper, nickel, molybdenum, tungsten, graphite, and quartz, is one of the most important uses of Nb and Ta. Thermal dissociation of $NbCl_5$, $NbBr_5$, NbI_5 , $TaCl_5$, $TaBr_5$, and TaI_5 yields metals of highest purity. The reduction of niobium and tantalum pentachlorides by hydrogen is at present the most economical manufacturing technique. Reduction of niobium and tantalum halides, combined with those of alloying admixtures, may open the way to creation of Nb- and Ta based Ti, Zr, Al, W, Mo, and Sn alloys. Thermal curves for the deposition of Nb and Ta-pentahalides obtained by Rolsten (Trans.

Card 1/2

ACCESSION NR: AP4017667

Metallurgical Soc. AIME, 215, No. 3, 472, 1952 and J. Electrochem Soc-y, 106, No. 11, 975, 1959) and the work of Yemel'yanov, Yevstyukhin, and Leont'yev (Sb. Metal-lurgiya i metallovedeniye chisty*kh metallov, no. 2, 27, 1960 and no. 3, 127, 1961) on the kinetics of niobium iodide refining are discussed. The latter identify 2 types of (temperature dependence for Nb-deposition — one monotonous at < 620 K and one with a maximum at > 620 K, which is believed to result from the existence of different Nb-iodides at 500-550 K (NbI₃) and 650 K (NbI₅). A maximum Nb-deposition rate of 30 g/hr was reached at 650 K in the reactor and 1135 K on the 1500 mm long thread. Orig. art. has: 5 graphs.

ASSOCIATION: Kafedra fiziko-khimicheskikh issledovaniy proizvodstva poluprovod-nikovy*kh materialov i chisty*kh metallov, Moskovskiy institut stali i splavov (Depart-ment of Physicochemical Studies on the Production of Semiconductors and Pure Metals, Moscow Institute for Steel and Alloys)

SUBMITTED: 20May63

DATE ACQ: 23Mar64

ENCL: 00

SUB CODE: ML

NO REF SOV: 011

OTHER: 021

2/2

Card

ACCESSION NR: AP4036963

S/0078/64/009/005/1049/1072

AUTHOR: Nisel'son, L. A.; Pustil'nik, A. I.; Sokolova, T. D.

TITLE: Orthobaric density and critical parameters of niobium and tantalum pentachlorides.

SOURCE: Zhurnal neorganicheskoy khimii, v. 9, no. 5, 1964, 1049-1072

TOPIC TAGS: niobium pentachloride, tantalum pentachloride, orthobaric density, critical parameter, critical density, critical pressure, critical temperature, niobium tantalum analysis, density temperature relationship, Berthelot equation, liquid vapor phase equilibrium, crystal liquid phase equilibrium

ABSTRACT: The orthobaric density of $NbCl_5$ and $TaCl_5$ throughout the liquid state and in the vapor state, and their critical parameters were determined (fig. 1). The densities of the liquid $TaCl_5$ and $NbCl_5$ and of their mixtures were measured precisely from their melting temperatures (216.2 and 204.2 C, respectively) to 300-320 C. The critical parameters for $NbCl_5$ were: critical temperature 534 C, density ρ_{crit} 0.68 g/cm³, pressure P_{crit} 46 atmospheres; for $TaCl_5$ were: 494 C, 0.89 g/cm³ and 43 atmospheres. Since the liquid-vapor phase and the crystal-

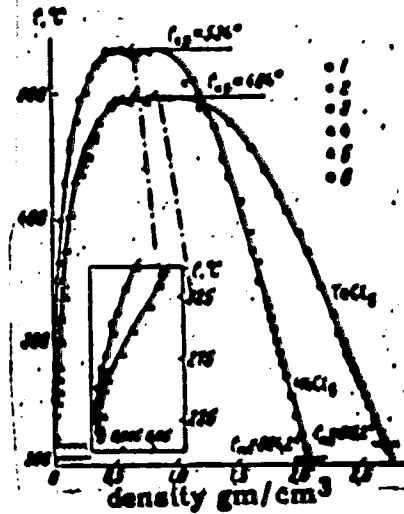
Card 1/3

ACCESSION NR: AP4036963

ENCLOSURE: 01

Fig. 1. Data for orthobaric density of NbCl₅ and TaCl₅.

1--NbCl₅ (vapor); 2--NbCl₅ (liquid), data obtained in a small picnometer;
 3--NbCl₅ (liquid), data obtained in large picnometer; 4--TaCl₅ (vapor);
 5--TaCl₅ (liquid), data obtained in small picnometer; 6--TaCl₅ (liquid), data obtained in large picnometer.
 t_{kp} = critical temperature
 t_{ms} = melting temperature



Card 3/3

NISEL'SON, L.A.; SOKOLOVA, T.D.

Orthobaric densities and the critical parameters of the niobium
and tantalum pentabromides. Zhur. neorg. khim. 9 no.9:2066-2067
8 '64. (MIRA 17:11)

1. Gosudarstvennyy nauchno-'sledovatel'skiy i proyektnyy
institut redkometallicheskoj promyshlennosti.

1-224841-85 EW(a)/EPT(a)/EPT(a)-2/EAT(b)/EAP(b) 17-1/Po-4 LJP(s) JI/W/JJ
ACCESSION NR: AP6020500 S/0076/54/009/012/2779/2730

AUTHOR: Nielsen, L. A. ; Stolberg, V. I.

RELATIVE volatility of zirconium tetrachloride and hafnium tetrachloride above their melting points

JOURNAL: Zhurnal neorganicheskoy khimii, v. 9, no. 12, 1984, 2730-2730

KEYWORDS: zirconium hafnium relative volatility, Rayleigh equilibrium evaporation, zirconium tetrachloride, hafnium tetrachloride, rectification process

ABSTRACT: Determination of such volatility is important, since the rectification process starts at such temperatures. The authors undertook direct determination of relative volatility of a mixture of both tetrachlorides at about 450C i.e. 15C above the melting point of ZrCl₄ by means of Rayleigh equilibrium evaporation. The concentration in the liquid stage of 0.13-0.15 mol-%. The equipment used and the procedure described, results are tabulated. Hafnium determination was conducted by spectral analysis after the zirconium tetrachloride had

L 23495-65

ACCESSION NR: AP500050

transformed into the dioxide. The mean value from 4 parallel tests gave
 2.70 ± 0.01 . Orig. art. has: 1 table and 1 figure

ASSOCIATION: None

EXAMINED: 05Aug63

ENCL: 00

SUB CODE: IC, OC

REF SOV: 004

OTHER: 002

Card 2/3

BR

ACCESSION NR: AP4024771

S/0000/64/007/003/0669/0672

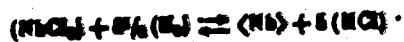
AUTHOR: Nisel'son, L. A.; Polyakov, Ya. N.; Krastovnikov, A. N.

TITLE: Research on the process of niobium extraction by reduction of NbCl₅ by hydrogen. II Communication II

SOURCE: Zhurnal prikladnoy khimii, v. 37, no. 3, 1964, 669-672

TOPIC TAGS: niobium extraction, reduction, NbCl₅, TaCl₅, equilibrium constant, NbCl₅ reduction

ABSTRACT: From an earlier work (Ya. N. Polyakov, L. A. Nisel'son, A. N. Krastovnikov, ZhPTKh, XXVI, 1, 25 (1963)), it follows that the reduction of NbCl₅ with hydrogen occurs more easily and at lower temperatures than the reduction of TaCl₅. From the data of temperature dependence of the equilibrium constant (Kp) of the reaction equilibrium yields (Fig. 1) and degree of conversion of



Card 1/3

ACCESSION NR: AP4024771

NbCl₅ into metal (Fig. 2) for various temperatures and NbCl₅ concentrations in the initial vapor-gaseous mixtures were computed. The data obtained makes it possible to estimate the temperature and concentration limits within which optimum operating conditions of the process can be created in industrial equipment, namely: 0.1-0.2 of niobium pentachloride mole in 1 mole of vapor-gaseous mixture and 1000-1300 C. In these conditions the rate of niobium precipitation was 0.7-1.5 g/cm². hr. with a yield of 1.5-3.2 g. of niobium in 1 mole of the mixture; degrees of conversion of niobium pentachloride into metal are 15-30% and specific consumption of electric power is 17-22 kw. hr. in one kg. of niobium (Fig. 3). It should be noted that the indexes mentioned above are not optimum and can be increased by taking into account the variation of the vapor-gaseous flow rate and apparatus design. "Graduate K. V. Tret'yakova took part in the experiment". Orig. art. has: 1 table, 5 figures.

ASSOCIATION: None

SUBMITTED: 28Dec62

DATE ACQ: 16Apr64

ENCL: 03

SUB CODE: 00

NO REF SOV: 001

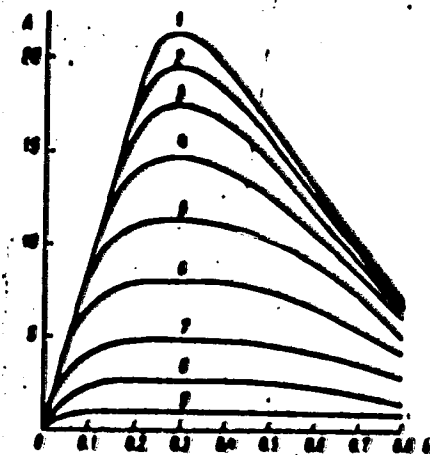
OTHER: 000

Card 2/5

ACCESSION NR: AP4024771

ENCLOSURE: 01

Fig. 1 - Dependence of equilibrium yield of niobium (A, g/mole of mixture) on initial concentration of niobium pentachloride (B, mole/mole of mixture) in vapor-gaseous mixture $\text{NbCl}_5 / \text{H}_2$ and on temperature. Temperature ($^{\circ}\text{K}$): 1 - 1500, 2 - 1400, 3 - 1300, 4 - 1200, 5 - 1100, 6 - 1000, 7 - 900, 8 - 800, 9 - 700.

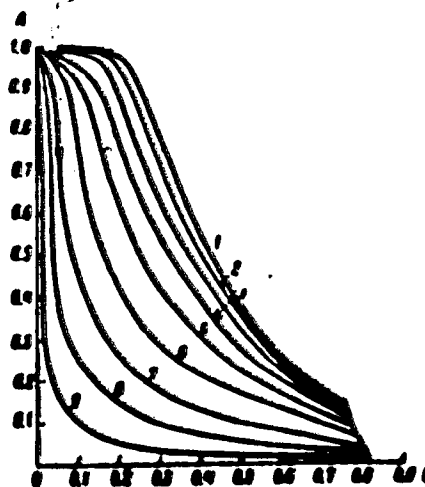


Card 3/5

ACCESSION NR: AP4024771

ENCLOSURE: 02

Fig. 2 - Dependence of equilibrium degree of conversion of niobium pentachloride into metal, on initial concentration of niobium pentachloride in vapor-gaseous mixture $\text{NbCl}_5 / \text{H}_2$ and on temperature, Symbols are the same as in Fig. 1

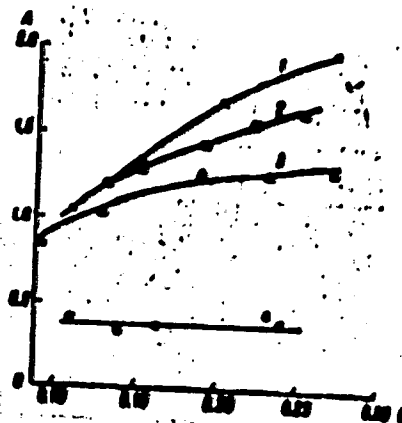


Card 4/5

ACCESSION NR: AF4024771

ENCLOSURE: 03

Fig. 3 - Dependence of specific electric energy consumption (A, kw. hr./kg.Nb) on temperature (B, °C) and concentration of niobium pentachloride in vapor-gaseous mixture NbCl₅ / H₂. Content NbCl₅ (mole): 1 - 0.1, 2 - 0.15, 3 - 0.20.



Card 5/5

L 61110-05 INT(n)/EXP(b)/INT(t) IJF(c) JD

ACCESSION NO: A73017469

U1/0370/65/001/003/004/007
649.2/8.049.6

AUTHORS: Flak'son, L. A.; Alaksyeva, N. N.; Ivanova, N. V.

12
B

TITLE: Purification of gallium by rectification of its trichloride

SOURCE: AN SSSR. Izvestiya. Metall., no. 3, 1965, 40-49

TOPIC TERMS: gallium trichloride, aluminum trichloride, refined gallium, rectification column, simple distillation, sieve plate column, packed column

ABSTRACT: The higher chloride $GaCl_3$ is an intermediate in the production of highly pure gallium by various methods such as extraction from HCl solutions, zone refining, and simple distillation. In this connection, the authors investigated the possibility of using $GaCl_3$ as the raw material for obtaining pure gallium by means

Card 1/1

APPROUSION NO: AP5017469

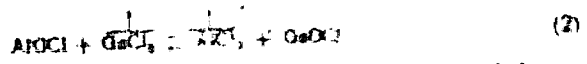
refined gallium (AlCl_3 0.36% by weight, FeCl_3 0.44%, PbCl_2 0.21%, CuCl_2 0.22%, and HgCl_2 0.26%) was prepared by fusion in an evacuated and soldered vial. Then one half of the mixture was subjected to simple distillation and the other, to rectification in a packed column of 20 mm diameter with a packing bed 390 mm high (glass rings - 4.5 mm in dia. per). As was expected, rectification proved to be more effective in isolating a purities than simple distillation. Thus, in these experiments, nearly 99% of gallium trichloride (in terms of the initial charge) was obtained with a content of $\text{Fe} > 1 \cdot 10^{-4}\%$, $\text{Pb} > 2 \cdot 10^{-6}\%$, $\text{Cu} > 4 \cdot 10^{-6}\%$. The isolation of aluminum was, however, less thorough: AlCl_3 is a much more volatile impurity than GaCl_3 , and therefore it is present in the very first fractions both during rectification and during distillation; at the same time, a large part of the aluminum remains in the still residue. Therefore a special series of experiments was performed to elucidate certain features of the behavior of aluminum during the distil-



Card 2/4

APPROVED FOR RELEASE: Tuesday, August 01, 2000
APPROXIMATE REF: AP5017469

The resulting aluminum oxychloride is nonvolatile. However, the results of the experiments with simple distillation indicate that even an excess of water does not convert all of the $AlCl_3$ to the nonvolatile form of the oxychloride. This is apparently related to the existence of a mobile equilibrium in the $GeCl_4$ salt



The findings on simple distillation indicate that reaction (2) is markedly shifted backward in the direction of the formation of $AlOCl$. Reaction (2) accounts for the... during the distillation and packed-column rectifica-

while the equilibrium of reaction (1) is the

Card 3/4

ACCESSION NO: AP5017469

which is more effectively removed from the reaction region. Thus, rectification is indeed a workable method of refining gallium. Orig. art. has: 5 figures, 6 tables, 5 formulas.

ASSOCIATION: none

ACQUIRED: 85 Jul 68

REF ID: A661004

EXCL: 00

SUB CODE: IC, EN

OTHER: 004

L 01798-66 EAT(m)/EPT(n)-2/EAP(t)/EAP(b) IJP(c) JD/AA/JG

ACCESSION NR: AP5021497

UR/0370/65/000/004/0097/0104
669.2/.8.049.6.296.297

AUTHOR: Nisel'son, L. A. (Moscow); Stol'yarov, V. I. (Moscow); Izhyanov, L. A. (Moscow); Korot'ev, YU. H. (Moscow)

TITLE: Separating zirconium and hafnium by fractionating their tetrachlorides

SOURCE: AN SSSR. Izvestiya. Metally, no. 4, 1965, 97-104

TOPIC TAGS: hafnium, zirconium, fractional distillation, metal purification

ABSTRACT: Mixtures of ZrCl₄ and HfCl₄ are experimentally separated by fractionation in metal columns with kilogram charges. The experimental equipment is shown in fig. 1 of the Enclosure. The results are tabulated and graphed. It was found that direct fractionation of the tetrachloride mixture is highly effective as a means for separating hafnium and zirconium. When the initial tetrachloride mixture contains 1.5-2.5% Hf, fractionation produces more than 50% Zr containing about 0.05% Hf. Up to 40% of the Hf in the original charge is concentrated in the head fractions with an average hafnium content of 20-25%. With initial hafnium contents of 16.5 and 13.5%, the maximum concentration of Hf in the head fractions of the

Card 1/3

L 01798-65

ACCESSION NR: AP5021897

distillate is 85.6 and 70.8% respectively. The experimental conditions produced a yield of 30-40 g/cm²·hr. Orig. art. has: 4 figures, 5 tables. O

ASSOCIATION: none

SUBMITTED: 25Jul64

ENCL: 01

SUB CODE: GC, MM

NO REF SOV: 007

OTHER: 002

Card 2/3

L 01798-66

ACCESSION NR: AP5021497

ENCLOSURE: 01

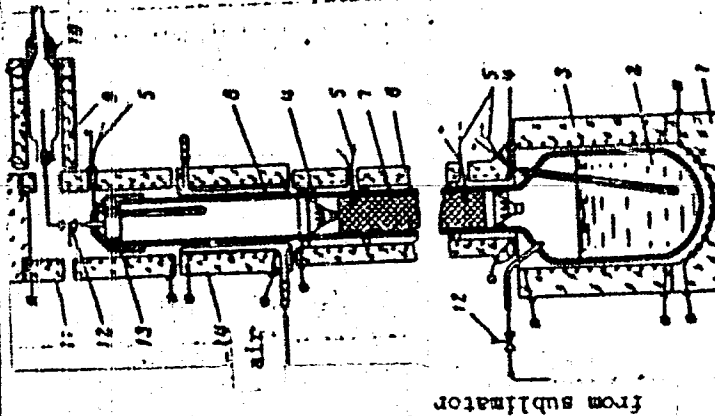


Fig. 1. Diagram of the fractionation column for separating a $ZrCl_4-HfCl_4$ mixture: 1--main electric heater for the still; 2--column still; 3--auxiliary electric heater for the still; 4--cone holding the packing; 5--thermocouples; 6--heat insulation for the column; 7--packing; 8--air-cooled sleeve for the fractionating column; 9--electric heater for the fraction receiver; 10--receiver for the hafnium fractions; 11--electric heater for the head section of the column; 12--needle valve; 13--head section of the fractionating column; 14--electric heater for the fractionating column

Card 3/3

PETUSEVICH, I.V. (Moskva); NISEL'SON, L.A. (Moskva); BELAYEV, A.I. (Moskva)

Obtaining titanium silicides by the simultaneous reduction
of titanium and silicon tetrachlorides by hydrogen. Izv. AN
SSSR. Mat. no.6:52-57 N-D '65. (MIRA 19:1)

1. Submitted October 14, 1964.

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ACQUISITION NUM: AX5002796

studied at temperatures ranging from the melting point to 110--1200C and the results are described by formulas. More exact melting points of pure MoCl and WCl were determined to be at 154.4C and 283C, respectively. Experimental results are given in tables 1, 2, and 3 of the Enclosure. The data given in this study are new and do not appear in the technical literature. Orig. art. has: 2 figures and 3 tables.

ORIGINATOR: Gosudarstvenny nauchno-issledovatel'skiy i proyektnyy institut
"Soyuznezh" Moscow (USSR) State Scientific

ACQUISITION NO: AP5002796

ENCLOSURE: 02

Table 2. Critical parameters and other properties of molybdenum pentachloride and tungsten hexachloride

Compound	Molecular weight, g	$t_m, ^\circ C$	$t_c - t_m, ^\circ C$	$P_c, g/cm^2$	$V_c, cm^3/mol$	P_c (est.), atms	ρ_{mole} for liquids, g/cm^3	Constants of Van Der Waals equation	
								$a \cdot 10^{-6}$ cm^5/mol	$b, cm^3/mol$
MoCl ₅	273.24	194.4	377.0	332.4	0.24	333.8	51.8	2.126	89.60
									198.2

Table 3. Viscosity of liquid molybdenum pentachloride and tungsten hexachloride

MoCl ₅		WCl ₆		MoCl ₅		WCl ₆	
t, °C	η , centipoise	t, °C	η , centipoise	t, °C	η , centipoise	t, °C	η , centipoise
110.5	0.856	285.5	1.193	270.0	0.664	342.3	0.880
221.0	0.750	290.5	1.161	280.5	0.628	331.5	0.814
230.5	0.732	308.7	1.042	311.5	0.479	370.7	0.742
240.5	0.687	309.2	1.051			385.7	0.693
253.5	0.624	320.1	0.972			430.3	0.630
264.0	0.579	323.0	0.963				

Cont 5/5

ANT(a)/EWP(b)/EWP(c) IIP(d) JD

REF ID: AFS008480

8/0078/65/010/003/0593/0595

16
15
14

Isel'bon, L. A.; Mogucheva, V. V.; Sokolova, T. D.

Critical parameters of phosphorus, arsenic, and antimony trichlorides

Journal neorganicheskoy khimii, v. 10, no. 5, 1965, pp. 576

Keywords: antimony trichloride, arsenic trichloride, phosphorus trichloride, temperature, critical density, orthobaric density

This study has been carried out because the available data on the substances concerned are disconnected and incomplete. Orthobaric density curves for SbCl₃, AsCl₃, and PCl₃ are plotted, and the critical temperatures and densities of the chlorides are obtained from the density data. The experimental data are processed by the method of least squares and presented in the form of interpolated equations. Densities of the SbCl₃ liquid from melting point to 500°C and of the AsCl₃ and PCl₃ from 0 to 120-140°C are measured with great accuracy. The results are given

temperatures for BiCl_3 and PCl_3 are also in good agreement with the available data.

REGISTRATION NH: AF5008480

is 20-30C higher for AsCl_3 . Orig. art has 1 formula, 2 figures, and 2

ORIG. SOURCE: Gosudarstvenny nauchno issledovatel'skiy i proyektnyy institut
metallicheskoy promyshlennosti (State Design and Planning Scientific Research
Institute of the Rare Metals Industry)

DATE: 11 Oct 63

ENCL: 02

SUB CODE: EC

REF ID: A66005

OTHER: 010

AF 5008480

ENCLOSURE: 01

Table 1. Orthobaric densities of antimony, arsenic, and phosphorus trichlorides

SbCl ₃		AsCl ₃		PCl ₃	
Vapor	Liquid	Vapor	Liquid	Vapor	Liquid
Temp, °C	Density, g/cm ³	Temp, °C	Density, g/cm ³	Temp, °C	Density, g/cm ³
182.4	0.00428	83.3	2.7332	186.5	0.0144
207.0	0.0043	78.4	2.5653	175.9	0.0239
247.5	0.0115	64	2.3727	169.3	0.0332
249.2	0.0108	123.1	2.2683	143.0	0.0578
259.3	0.0160	153.5	2.1875	125.4	0.0909
289.7	0.0225	182.7	2.1281	115.8	0.135
296.3	0.0242	207.7	2.0703	101.5	0.1815
		232.0	2.0142	82.7	0.243

011.5 | 0.0001 | 201.5 | 12.1100 | 153.2 | 0.773 | 1128.5711 | 91508.458.1 | 87.183 | 7408.4 | 1.74009

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EXHIBITION NO: AF5008480

ENCLOSURE: 02

Page 1 cont.

113.5	0.0354	322.5	2.08	378.9	0.412	148.7711	4071.25	7.43	158	113.8711	3866
118.7	0.0419	366.0	2.04	378.5	0.454	148.8711	4716.29	0.01	203	140.0	1.34
114.5	0.0406	374.7	1.94	381.2	0.360	152.5	1.81	27	8	0.715	400.0
113.0	0.037	418.0	1.87	382.2	0.272	152.5	1.81	27	8	0.715	400.0

412.8	0.238	528.8	1.19	294.0	1.57	234.5	1.07
411.8	0.238			290.8	1.49	247.9	1.02
412.8	0.444			319.5	1.38	251.5	1.00
415.7	0.441			317.5	1.22	254.0	0.99
419.9	0.555			348.0	1.23	240.0	0.95
431.5	0.705			353.0	1.20	239.5	0.90
432.8	0.792			351.2	0.80		
431.0	0.819			382.5	0.88		
440.9	0.858						

The accurate density measurements were obtained in a pycnometer of large volume.

... ..
... ..
... ..

11137: Density, viscosity, and surface tension of silicon tetrachloride and trichloro-
silane

RUSSIAN SOURCE: Zhurnal neorganicheskoy khimii, v. 10, no. 4, 1965, 1297-1299

ENGLISH TAGS: silicon tetrachloride, trichlorosilane, chloride density, chloride viscosity, chloride surface tension

ABSTRACT: The article contains a series of data on the physical properties of silicon tetrachloride and trichlorosilane, two important source materials for the production of high-purity silicon. It compares the results obtained in the literature with the author's. In this report, the authors present a series of measurements of the density, viscosity, and surface tension of $SiCl_4$ and $SiCl_3H$ at various temperatures slightly above their normal boiling points. The chlorides studied were thoroughly purified by chemical means and by distillation. Density was measured in quartz pycnometers, viscosity in a capillary viscometer, and surface tension by the method of maximum bubble pressure in a bubble. All the measurements were carried out in sealed devices in order

25-45

EXENSION NR: AP5015012

include the adverse effect of moisture. The data obtained are tabulated and illustrated with graphs; they were also treated by the method of least squares, and are expressed in the form of exponential interpolation equations. (Orig. art. has 2 figures and 6 tables.)

ASSOCIATION: None

SUBMITTED: 03Aug63

ENCL: 00

SUB CODE: IC

NO REF SDV: 008

OTHER: 008

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6, 180-65

REF(a), ZVPR 1 547

1965 1519

ACQUISITION NO. AP5018241

BR/0078/65/010/007 1516/1519

546.6.3.131+546.681.3.131

22
12

AUTHOR: Nival'son, L. A., Sokolova, T. D.

TITLE: Density, viscosity, and surface tension of aluminum and gallium trichloride

SOURCE: Zhurnal neorganicheskoy khimii, v. 10, no. 7, 1965, 1516-1519

DESCRIPTORS: Aluminum chloride; gas; gallium trichloride; density; viscosity; surface tension

ABSTRACT: Pycnometric measurements of density yielded the following equations:

$\rho_{Al_2Cl_6} = 1.2641 - 2.32 \times 10^{-5} \Delta t - 1.16 \cdot 10^{-6} \Delta t^2$ g/cm³ from $t_{fus} = 192.5$ to 216.0C, where $\Delta t = t - 192.50$ (mean square error $\Delta \rho_{Al_2Cl_6} = 0.0004$), and

$\rho_{Ga_2Cl_6} = 2.0546 - 1.985 \times 10^{-5} \Delta t - 1.04 \times 10^{-6} \Delta t^2$ g/cm³ from $t_{fus} = 78.0$ to 240.0C, where $\Delta t = t - 78.00$ (mean square error $\Delta \rho_{Ga_2Cl_6} = 0.0013$).

From the peaks of orthobaric curves, the critical temperatures (t_{cr}) were found; the critical densities were also determined. From viscosimetric data, the following equations were derived:

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$\eta_{Al_2Cl_6} = 3.002 \times 10^{-1} - 2.899 \times 10^{-3} \Delta t + 1.232 \times 10^{-5} \Delta t^2$ poise from $t_{fus} = 192.5$ to 276.00 , where $\Delta t = t - 192.50$ (mean square error $\Delta \eta_{sq} = 0.003$),

$\eta_{Ca_2Cl_6} = 1.768 - 2.22 \times 10^{-2} \Delta t + 1.47 \times 10^{-4} \Delta t^2 - 3.74 \times 10^{-7} \Delta t^3$ poise from $t_{fus} = 78.0$ to 247.00 , where $\Delta t = t - 78.0$ (mean square error $\Delta \eta_{sq} = 0.013$). The data obtained for the surface tension can be represented by the following equations:

$\sigma_{Al_2Cl_6} = 9.77 - 7.33 \times 10^{-2} \Delta t$ dyn/cm from $t_{fus} = 192.5$ to 2850 , where $\Delta t = t - 192.50$ (mean square error $\Delta \sigma_{sq} = 0.1$), and

$\sigma_{Ca_2Cl_6} = 23.9 - 1.0 \times 10^{-1} \Delta t + 8.7 \times 10^{-3} \Delta t^2$ dyn/cm from $t_{fus} = 78.0$ to 300.00 , where $\Delta t = t - 78.00$ (mean square error $\Delta \sigma_{sq} = 0.3$). Orig. art. has: 3 figures, 6 formulas and 4 tables.

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Card 2/2 *AP*

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TITLE: Liquid-vapor equilibria in systems formed by silicon tetrachloride and silicon trichloride with dibutyl ether and some additives

SOURCE: Zhurnal fizicheskoy khimii, v. 39, no. 1, 1965, 161-165

TOPIC TAGS: equilibrium condition, silicon tetrachloride, silicane trichloride, dibutyl ether

ABSTRACT: Experiments were performed on equilibria between liquids and vapors in systems formed by SiCl_4 and SiHCl_3 with dibutyl ether and some added chlorides. Graphs show the dependence of the logarithms of vapor pressures on the inverse absolute temperature in mixtures of SiCl_4 and SiHCl_3 with dibutyl ether, liquid-vapor phase equilibria in SiCl_4 and SiHCl_3 with dibutyl ether at 760 mm (with a negative deviation from Raoult's law), and also the dependence between the coefficient of activity and the composition of the liquid phase. Tables present the coefficients of $\log p = A - B/T$ equation for SiCl_4 - and SiHCl_3 -dibutyl ether systems. Also the relative volatility in SiCl_4 and SiHCl_3 systems with added